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SOLVENT EXTRACT AND THE PLASTIC PROPERTIES OF COAL

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ABSTRACT

The amount of material extractable from coal by certain solvents is related to the maximum fluidity of the coal as measured by the Gieseler plastometer, according to earlier work by Pierron, Rees, and Clark. This relationship has now been further studied for additional coals of different ranks and levels of fluidity. In addition some attention has been given to recombining extracts with residues in different proportions and determining Gieseler fluidities of these blends. Data are presented in this report showing that, for the coals studied, yields of extracts are proportional to maximum fluidities although not necessarily directly proportional. Further data presented indicate that return of extracts to inert residues restores fluidity beyond that of the original coal.

INTRODUCTION

The amount of material that can be extracted from coal using certain solvents is related to the maximum fluidity of the coal as measured by the Gieseler plastometer, according to previous work by Pierron, Rees, and Clark (1959). Although the extracts exhibited plastic properties, the resultant coal residues showed no such properties in the plastometer. However, when the extract was recombined with the residue, plasticity not only was restored but the maximum fluidity was greater than that of the original coal.

Because the earlier work had covered studies on only three samples of coal, we decided to extend it to cover more coals of various ranks, volatile matter contents, and maximum fluidity levels. This report presents the results of these extended studies. In this work emphasis was placed on the relationship of quantity of extract to maximum fluidity and on the maximum fluidities of blends made by combining extracts and residues in different proportions. Chemical analyses for coals, extracts, and residues are presented, and, in addition, molecular weight data are given for the pyridine extracts and fractions thereof derived by further solvent treatment.

Acknowledgments

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EXPERIMENTAL PROCEDURE

Samples

The different coals investigated are identified by code letters for future reference in the manuscript and described as follows:

Coal:

A Oxidized Illinois high-volatile B bituminous, No. 5 Coal, Saline County
B Nonoxidized Illinois high-volatile B bituminous, No. 5 Coal, Saline County
C Illinois high-volatile B bituminous, No. 5 Coal, Gallatin County
D Illinois high-volatile A bituminous, Willis Coal, Gallatin County
E Eastern high-volatile A bituminous, Hernshaw Seam, West Virginia
F Eastern high-volatile A bituminous coal, Eagle Seam, West Virginia
G Eastern medium-volatile bituminous coal, Jewell Seam, Virginia
H Eastern low-volatile bituminous coal, Pocahontas No. 3 Seam, West Virginia

With the exception of Coal A, all samples were obtained from the mines or other sources in a size of 2 by 3 inches or larger. They were reduced to less than one-fourth inch by means of a jaw crusher, and air-dried overnight at 34°C. The air-dried samples were then stage ground, producing a minimum amount of fines, to pass a 40-mesh sieve. The minus 60-mesh portions were discarded. The minus 40 plus 60-mesh stock samples so obtained were not representative of the coals investigated but were prepared specimens that permitted a better basis for comparison. As previously reported by Pierron et al. (1959), coal A was obtained by subjecting a portion of coal B to air oxidation until the plastic properties were completely destroyed.

To minimize the effect of oxidation the samples were stored under an oxygen-free nitrogen atmosphere.

Solvent Extractions

Each sample was extracted according to the scheme and procedure described by Pierron et al. (1959).

Briefly, the procedure was as follows:

Ten grams of the stock sample was digested for six hours with 150 ml of anhydrous pyridine at 114°C., the residue was separated from solvent plus extract by filtration, washed, and dried for two hours under vacuum at 116°C. The pyridine extract was obtained by removal of the solvent in a rotating vacuum evaporator. In order to obtain enough extract the pyridine digestion was carried out several times. The yield of the pyridine extract was determined after drying under vacuum for two hours at 100°C.

Ten grams of pyridine extract was then extracted for six hours with 150 ml of chloroform at 61°C. The residue was separated from solvent plus extract by filtration, washed, and dried for three hours under vacuum at room temperature. The extract was separated from the solvent by evaporating the chloroform on a steam bath at atmospheric pressure; it was then dried.

A weighed amount of the chloroform extract (1 to 2 grams) was extracted for six hours with 150 ml of n-hexane at 67°C. The extract and residue were obtained and dried following the procedure described for the chloroform extraction.

To minimize the effect of oxidation, the material was extracted, filtered, and stored under an oxygen-free nitrogen atmosphere.

Analytical Data

Because the amounts of extracts obtained were so small, micro techniques were used for the ultimate analyses of extracts and residues. The ultimate analysis for each coal also was made by micro technique.

Proximate and ultimate analysis data, calorific values, and yields of extracts are reported on a moisture- and ash-free basis.

Plasticity measurements of coals, pyridine extracts, and residues were made by a Gieseler plastometer as modified and described by Rees and Pierron (1954). Each determination was made in duplicate and the average was reported.

The ebullioscopic method for molecular weight determination was used. Each determination was made in triplicate, and the average reported. The solvents were as follows: pyridine for pyridine extracts and chloroform residues; chloroform for chloroform extracts and n-hexane residues; and n-hexane for n-hexane extracts.

RESULTS

Table 1 gives the chemical analyses, calorific values, and yields of extracts on a moisture- and ash-free basis, the free swelling indexes, and the Gieseler plasticity data for the eight series of samples investigated.

Table 2 shows the petrographic analyses of the specially prepared stock samples of coals investigated. The designation and measurement of the macerals is in accordance with the petrographic procedures reported by Marshall et al. (1958).

Table 3 summarizes for each series the ultimate analysis data obtained by micro methods on a moisture- and ash-free basis, the molecular weights, and the atomic H/C and O/C ratios.

Table 4 summarizes for each series the molecular weights, atomic H/C and O/C ratios of pyridine, chloroform, and n-hexane extracts.

Table 5 shows the free swelling indexes and Gieseler plasticity data for Coal C, its pyridine residue, pyridine extract, and a series of blends. Compositions of the blends range from a mixture of pyridine extract and residue in proportions that duplicate the original percentage composition of the coal to a mixture containing only 1 percent extract.

Table 1. - Chemical Analyses (M-A free), Free Swelling Indexes, Gieseler Plasticity Data, and Yields of Extract (M-A free coal basis) of the Coals Investigated

	Coals*							
	A	B	C	D	E	F	G	H
Proximate**								
Volatile matter	40.5	40.3	43.6	41.0	41.3	32.1	23.3	18.5
Fixed carbon	59.5	59.7	56.4	59.0	58.7	67.9	76.7	81.5
Ultimate†								
Hydrogen	5.95	5.85	5.28	5.47	5.93	5.28	5.27	5.68
Carbon	82.49	83.18	79.17	85.02	86.66	87.97	89.68	91.21
Nitrogen	1.59	2.02	1.82	1.58	1.34	1.63	.86	1.12
Oxygen	8.50	7.25	6.67	6.09	5.13	4.68	3.61	1.11
Sulfur	1.50	1.70	7.06	1.84	.90	.44	.58	.88
Calorific value** Btu/lb.	14579	14658	14574	15442	15510	15427	15786	15736
Free swelling index**	1	6	7	1	5½	5	9	9
Gieseler plasticity								
Softening temp. C. (0.5 dial div./min.)		384	375	390	364	392	405	447
Fusion temp. C. (5.0 dial div./min.)		406	399	442	395	415	426	480
Max. fluidity temp. C.		431	428	442	430	455	469	480
Solidification temp. C.		462	474	480	485	494	507	510
Max. fluidity (dial div./min.)	0	80	3,500	5	37,500	6,900	2,083	5
Yield of extract in percent								
Pyridine extract	13.6	20.7	22.2	17.6	23.8	20.4	18.3	14.2
Chloroform extract	5.1	5.8	7.8	5.5	9.0	8.2	7.6	5.4
n-hexane extract	.7	2.4	3.0	1.4	3.4	3.2	2.9	1.3

*Identification of coals:

A. Oxidized, Illinois high-volatile B bituminous, No. 5 Coal, Saline County.
B. Non-oxidized, Illinois high-volatile B bituminous, No.5 Coal, Saline County.
C. Illinois high-volatile B bituminous, No. 5 Coal, Gallatin County.
D. Illinois high-volatile A bituminous, Willis Coal, Gallatin County.
E. Eastern high-volatile A bituminous, Hernshaw Seam, West Virginia.
F. Eastern high-volatile A bituminous coal, Eagle Seam, West Virginia.
G. Eastern medium-volatile bituminous coal, Jewell Seam, Virginia.
H. Eastern low-volatile bituminous coal, Pocahontas No. 3 Seam, West Virginia.

** ASTM methods.

† Micro methods.

Table 2. - Petrographic Analyses of Specially Prepared Coal Samples

Coals*	Maceral Proportions - percent			
	Vitrinite	Exinite	Inertinite	Visible mineral matter
C	86.6	3.5	5.5	4.4
D	31.7	23.6	43.2	1.5
F	58.4	11.6	28.2	1.8
G	82.8	.6	16.3	.3
H	84.8	.2	14.5	.5

*Coals:

C - Illinois high-volatile B bituminous, No. 5 Coal, Gallatin County.
D - Illinois high-volatile A bituminous, Willis Coal, Gallatin County.
F - Eastern high-volatile A bituminous coal, Eagle Seam, West Virginia.
G - Eastern medium-volatile bituminous coal, Jewell Seam, Virginia.
H - Eastern low-volatile bituminous coal, Pocahontas No. 3 seam, West Virginia.

Table 3. - Ultimate Analyses by Micro Methods (M-A free) Basis, Molecular Weights, and Atomic H/C and O/C Ratios for the Coals Investigated

	Coal	Pyridine		Chloroform		n-hexane	
		Extract	Residue	Extract	Residue	Extract	Residue
COAL A Oxidized Illinois High-volatile B Bituminous, No. 5 Coal, Saline County							
Hydrogen	5.95	5.95	5.42	7.11	5.32	8.30	7.28
Carbon	82.49	83.91	81.66	84.85	80.72	87.08	84.82
Nitrogen	1.56	1.61	1.45	1.63	1.65	1.54	3.69
Oxygen	8.50	7.70	10.30	5.61	11.74	2.29	3.59
Sulfur	1.50	.83	1.17	.80	.57	.79	.62
Molecular weight		1600		490	700	380	550
Atomic H/C	.859	.844	.791	.999	.786	1.135	1.023
Atomic O/C	.077	.069	.094	.050	.100	.019	.031
COAL B Nonoxidized Illinois High-volatile B Bituminous, No. 5 Coal, Saline County							
Hydrogen	5.85	5.99	5.53	7.12	5.37	7.85	7.92
Carbon	83.18	84.02	81.91	84.76	79.45	87.27	86.32
Nitrogen	2.02	2.10	2.13	2.25	2.25	.56	2.42
Oxygen	7.25	7.17	9.04	4.80	11.70	3.32	2.67
Sulfur	1.70	.72	1.39	1.07	1.23	1.00	.67
Molecular weight		1500		475	600	362	520
Atomic H/C	.837	.849	.805	1.000	.805	1.072	1.093
Atomic O/C	.065	.064	.083	.042	.110	.029	.024
COAL C Illinois High-volatile B Bituminous, No. 5 Coal, Gallatin County							
Hydrogen	5.28	5.87	5.54	7.63	5.81	9.20	6.27
Carbon	79.17	82.76	77.71	85.34	80.24	87.25	86.36
Nitrogen	1.82	1.88	2.10	1.83	1.95	.83	2.39

Table 3 (continued)

	Coal	Pyridine Extract	Pyridine Residue	Chloroform Extract	Chloroform Residue	n-hexane Extract	n-hexane Residue
COAL C (continued)							
Oxygen	6.67	7.59	8.19	3.38	9.91	2.44	4.42
Sulfur	7.06	1.90	6.46	1.82	2.09	.28	.56
Molecular weight		1650		480	685	390	540
Atomic H/C	.801	.852	.856	1.073	.870	1.267	.872
Atomic O/C	.064	.068	.079	.030	.093	.021	.039
COAL D Illinois High-volatile A Bituminous, Willis Coal, Gallatin County							
Hydrogen	5.47	6.15	5.48	6.21	5.93	9.04	7.08
Carbon	85.02	85.86	85.15	85.43	84.11	87.42	86.62
Nitrogen	1.58	1.98	1.76	1.82	1.47	1.10	1.59
Oxygen	6.09	5.39	6.69	5.96	7.90	2.15	4.02
Sulfur	1.84	.62	.92	.58	.59	.29	.69
Molecular weight		1680		520	800	415	682
Atomic H/C	.773	.860	.773	.873	.847	1.242	.982
Atomic O/C	.054	.048	.059	.052	.070	.018	.035
COAL E Eastern High-volatile A Bituminous, Hernshaw Seam, West Virginia							
Hydrogen	5.93	6.42	5.43	6.70	5.95	8.58	6.88
Carbon	86.66	86.75	85.67	87.64	82.76	87.99	88.45
Nitrogen	1.34	1.96	1.49	1.32	1.86	1.32	2.00
Oxygen	5.13	4.14	6.33	3.29	8.60	1.59	1.90
Sulfur	.90	.73	1.08	1.05	.83	.52	.77
Molecular weight		1740		565	895	447	710
Atomic H/C	.814	.882	.756	.911	.856	1.161	.928
Atomic O/C	.044	.036	.056	.029	.078	.014	.016
COAL F Eastern High-volatile A Bituminous Coal, Eagle Seam, West Virginia							
Hydrogen	5.28	5.96	5.03	7.33	5.16	8.57	7.15
Carbon	87.97	87.63	87.18	89.23	84.45	87.23	88.76
Nitrogen	1.63	1.58	1.21	1.43	.90	.85	1.45
Oxygen	4.68	4.46	6.21	1.50	9.19	3.23	2.29
Sulfur	.44	.37	.37	.51	.30	.12	.35
Molecular weight		1650		550	887	471	750
Atomic H/C	.721	.816	.693	.987	.734	1.180	.968
Atomic O/C	.040	.038	.054	.012	.081	.027	.019
COAL G Eastern Medium-volatile Bituminous Coal, Jewell Seam, Virginia							
Hydrogen	5.27	6.55	5.40	8.37	5.67	8.93	6.93
Carbon	89.68	86.53	89.35	87.82	84.32	87.41	87.43
Nitrogen	.86	1.41	1.14	1.54	1.47	.85	1.82
Oxygen	3.61	4.90	3.63	1.70	7.84	2.42	3.14
Sulfur	.58	.61	.48	.57	.70	.39	.68
Molecular weight		1400		570	890	490	724
Atomic H/C	.705	.910	.725	1.145	.807	1.226	.952
Atomic O/C	.031	.043	.031	.015	.069	.021	.027

Table 3 (continued)

	Coal	Pyridine Extract	Pyridine Residue	Chloroform Extract	Chloroform Residue	n-hexane Extract	n-hexane Residue
COAL H							
Eastern Low-volatile Bituminous Coal, Pocahontas No. 3 Seam, West Virginia							
Hydrogen	5.68	6.41	5.34	6.92	5.47	8.70	6.92
Carbon	91.21	86.20	91.47	87.91	83.61	86.97	86.85
Nitrogen	1.12	1.26	1.30	1.84	1.47	.70	2.20
Oxygen	1.11	5.81	1.15	2.93	8.94	3.37	3.51
Sulfur	.88	.32	.74	.40	.51	.26	.52
Molecular weight		1175		575	899	525	774
Atomic H/C	.748	.893	.701	.945	.786	1.202	.957
Atomic O/C	.009	.050	.009	.025	.080	.029	.030

Table 4. - Molecular Weight, Atomic H/C and O/C Ratios of Pyridine, Chloroform, and n-hexane Extracts for the Coals Investigated

Coal	Pyridine Mol. wt.	Pyridine Atomic H/C	Pyridine Atomic O/C	Chloroform Mol. wt	Chloroform Atomic H/C	Chloroform Atomic O/C	n-hexane Mol. wt.	n-hexane Atomic H/C	n-hexane Atomic O/C
A	1600	.844	.069	490	.999	.050	380	1.135	.019
B	1500	.849	.064	475	1.000	.042	362	1.072	.029
C	1650	.852	.068	480	1.073	.030	390	1.267	.021
D	1680	.860	.048	520	.873	.053	415	1.242	.018
E	1740	.882	.036	565	.911	.029	447	1.161	.014
F	1650	.816	.038	550	.987	.012	471	1.180	.027
G	1400	.910	.043	570	1.145	.015	490	1.226	.021
H	1175	.893	.050	575	.945	.025	525	1.202	.029

Table 5. - Free Swelling Indexes, Gieseler Plasticity Data of Coal C, Pyridine Residue, Extract and Blends*

Illinois High-volatile B Bituminous, No. 5 Coal, Gallatin County

	Free swelling index	Softening	Fusion	Max. fluid.	Setting	Plastic range	Max. fluid dial div. per min.	Remarks**
		Gieseler plasticity temperatures in °C.						
Coal C	7	375	399	428	474	99	3,500	(1)
Pyridine residue	1	none	none	none	none	none	0	(2)
Pyridine extract	> 9	350	385	420	472	125	> 37,500	(3)
18.3% Extract + 81.7% residue†	9	335	387	422	475	120	12,600	(4)
15.0% Extract + 85.0% residue	8 1/2	360	391	425	475	115	10,700	(4)
10.0% Extract + 90.0% residue	7 1/2	367	394	426	474	107	8,000	(4)
5.0% Extract + 95.0% residue	6	380	402	435	480	100	1,700	(5)
1.0% Extract + 99.0% residue	2	382	405	433	481	99	62	(6)

* Blends made up on basis of air-dry values. † Made up to duplicate original coal.

** (1) Coal swells to ½ height Gieseler barrel.
(2) Residue can be poured from Gieseler crucible.
(3) Pyridine extract swells out of crucible.
(4) Blends swell to 2/3 height Gieseler barrel.
(5) Swelling equals the swelling of original coal.
(6) In the plastic state this blend does not swell.

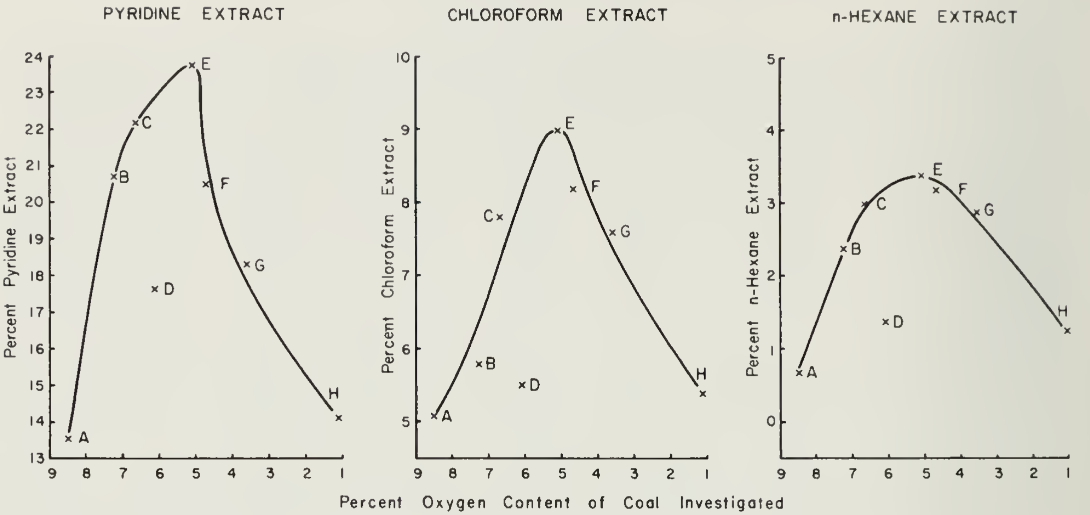

Fig. 1. - Yields of extracts versus oxygen content of the coals investigated.

DISCUSSION

The following discussion is limited to the eight specially prepared samples of coal. The original samples were selected to represent a progressive increase in rank from high-volatile B bituminous to low-volatile bituminous coal. It must be emphasized that to project similar conclusions to other coals may be misleading.

In table 1, the samples A to H are arranged in order of decreasing oxygen content. Figure 1, plotted on a linear scale, demonstrates the relationship between yields of pyridine, chloroform, and n-hexane extracts and amount of oxygen in the coals investigated. The amount of oxygen is shown as decreasing to conform, in general, with the increase in rank. With the exception of the D or Willis Coal, the samples indicate that as the oxygen content progressively decreases, the percentage of extracts increases to a maximum, then decreases. As compared to the chloroform and n-hexane extract, the curve for pyridine extract is unsymmetrical. This will be discussed later.

Figure 2 shows the relationship between the progressively decreasing volatile matter and oxygen contents of the coals investigated and their maximum Gieseler fluidities. The volatile matter and oxygen contents are plotted on a linear scale and the fluidities on a logarithmic scale. Here, as in figure 1, the point representing sample D or Willis Coal does not appear to follow the general relationship for oxygen versus fluidity.

It may be added that, in spite of possible error reflected in the calculation of oxygen in the ultimate analysis of coal, the curve shown in figure 1 appears to be symmetrical and indicates that as the amount of oxygen in coal decreases, the maximum Gieseler fluidity increases to a maximum, then decreases. In contrast, the Gieseler maximum fluidity of coal is independent of the volatile matter content if the volatile matter is greater than 39 percent on a moisture- and ash-free basis. For volatile matter less than 39 percent, it appears that as the volatile matter decreases (or rank increases), the Gieseler maximum fluidity decreases.

Figure 3 shows the yields of pyridine extracts as a function of the Gieseler maximum fluidities for the eight coals investigated. The decreasing yields of extracts are plotted on a linear scale and the maximum fluidities on a logarithmic scale. As the rank of the coal increases the yields of pyridine extract increase and maximum fluidities increase up to a maximum; then, as the rank continues to increase, the yield of extract and maximum fluidity decrease. Both the ascending and descending parts of the curve appear to approach a straight-line relationship.

Figure 4 shows the yields of chloroform and n-hexane extracts as a function of the Gieseler maximum fluidities. The data are plotted on a scale similar to that of figure 3. Attention is called to the fact that a more direct relationship is apparent between the chloroform and n-hexane extracts and their maximum fluidities than between the pyridine extracts and their fluidities.

During the pyridine extraction procedure it was noted that the amount of swelling of the pyridine residue progressively decreased as rank of coal increased. For example, the dried pyridine residue of Coal B had a volume approximately twice as great as

Fig. 3. - Maximum Gieseler fluidity versus percentage of pyridine extract.

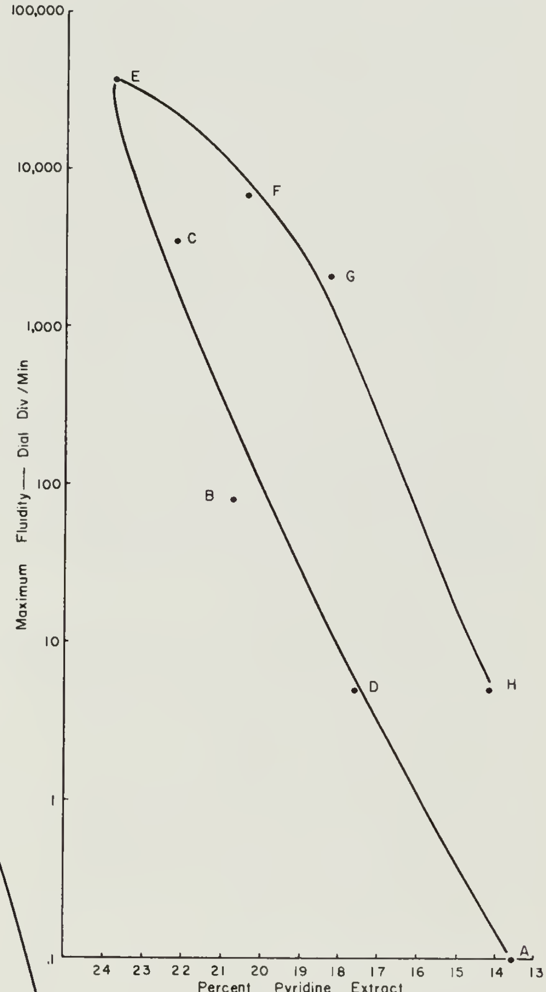

Fig. 2. - Maximum Gieseler fluidities versus volatile matter and oxygen contents.

that of its parent coal. In contrast, the volume of the dried pyridine residues of coals F, G, and H appeared to be similar to those of the original coals.

It might be postulated that as swelling of coal in contact with pyridine becomes greater, the probability of dispersion of coal particles in the solvent would increase. This behavior has been previously reported and discussed by Pierron et al. (1959). Such an assumption also is indicated by comparing the shape of the pyridine extract curve in figure 1 to those of the chloroform and n-hexane curves. If the interaction of pyridine with coal caused only solution with no colloidal dispersion of coal particles, it might be expected that the pyridine extract values for samples B and C would be lower and thus would contribute to a more symmetrical curve.

Pursuing this line of thinking with reference to figure 3, it is interesting to note that if the determined yields of pyridine extract for coals B and C are high by approximately 4 percent and 3 percent respectively, due to colloidally dispersed coal particles, the values would fall on the right-hand portion of the curve thus permitting construction of a single curve without a doubled back portion. In other words it would indicate a more direct relation between yields of pyridine extract and maximum fluidity regardless of the rank of coals studied. Point D in figure 3 would not fall on the single curve so constructed, but this might be explained by the fact that sample D was shown to contain a considerably larger amount of inertinite than the other coals (table 2).

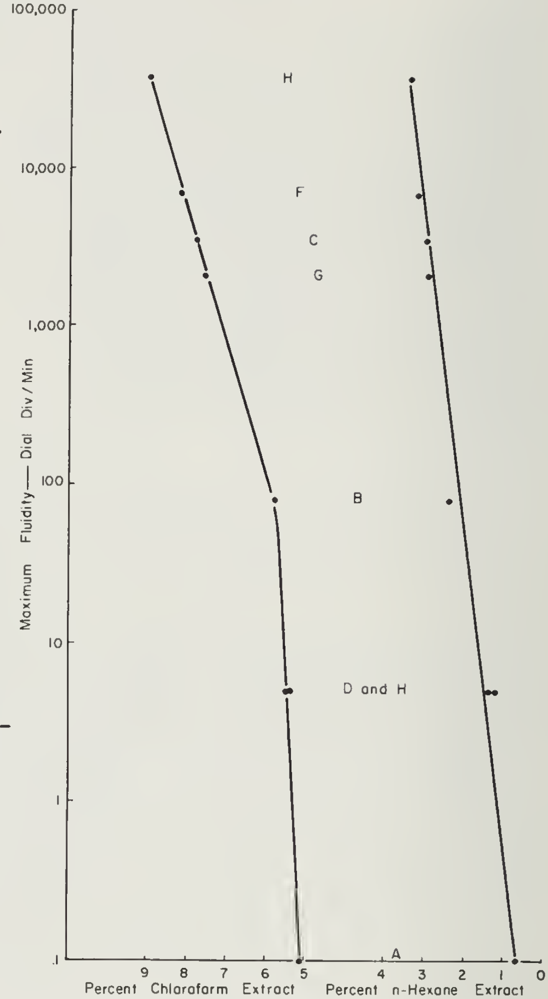

Fig. 4. - Maximum Gieseler fluidity versus percentage of chloroform and n-hexane extracts.

Table 4 compares the molecular weights and atomic H/C and O/C ratios of pyridine, chloroform, and n-hexane extracts for the coals investigated. As the rank of the coals increases from sample A to sample H, the H/C and O/C ratios of the pyridine extracts remain more or less constant, but the molecular weights decrease from 1600 to 1175. Such decrease in molecular weight may be related to the lesser amount of dispersed particles of the parent coal in the extract as the rank increases. The chloroform extracts indicate similar H/C and O/C ratios, but as rank increases the molecular weights increase from 490 to 575. The n-hexane extracts show similar H/C and O/C ratios, and as in the case of chloroform extracts, as rank increases molecular weights increase from 380 to 525.

Table 5 shows free swelling indexes, Gieseler plasticity data of coal C, pyridine residue and extract, and blends made of extract and residue. A blend of

18.3 percent pyridine extract and 81.7 percent residue was made to duplicate the original percentage composition of the coal on an air-dried basis. Other blends were made up of respectively 15, 10, 5, and 1 percent extract and 85, 90, 95 and 99 percent residue. An addition of 5 percent of extract to the residue increased the free swelling index from 1 to 6 or to a value approximately equivalent to that of the original coal.

The progressive addition of extract to residue progressively lowered the softening and fusion temperatures, had little effect on the maximum and setting temperatures, and increased the plastic ranges and maximum fluidities. Stephens (1958) reported similar results when pitch was added to coal. Nevertheless it appears that addition of 5 or 10 percent pyridine extract to residue induces plastic properties in a temperature range similar to the plastic temperature range of the original coals but with considerably greater maximum fluidities than those of the original coals.

CONCLUSIONS

1) Data are presented to indicate that yields of extract obtained with each solvent increase with decrease of oxygen content of samples to a maximum and then decrease with further decrease of oxygen content. Attention is called to the possibility that extract yields for certain of the coals possibly may have been high due to colloidal dispersion of coal in the solvent.

2) Yields of extracts for each solvent are proportional, although not necessarily directly proportional, to fluidities of the coals as measured by the Gieseler plastometer.

3) For the eight coals studied, the pyridine, chloroform, and n-hexane extracts for each coal show similar analyses.

4) The data presented indicate that fluidity is related to the quantity of extract obtained rather than to difference in kind of extract.

5) Addition of pyridine extract to nonplastic residue induces fluidity higher than that of the original coal.

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114